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PROCESSING - PLATING SYSTEMS - UNCLAD 2020
ALUMINUM ALLOY - CHEMICAL AND PHYSICAL
PROPERTIES - EVALUATION OF

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 (FORT WORTH)

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TEST NO. F-7882
 MODEL B-58

REPORT FGT-2407

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TITLE

PROCESSING - PLATING SYSTEMS - UNCLAD 2020 ALUMINUM ALLOY -
CHEMICAL AND PHYSICAL PROPERTIES - EVALUATION OF -

SUBMITTED UNDER
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The tests described in this report were conducted between
 March 15, 1958, and September 10, 1959.

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GROUP: CHEMISTRY LABORATORY
 ENGINEERING TEST LABORATORIES

REFERENCE: FGT-2106, FPS-0017,
-0028, -0029, -0045 & -0060

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D. C. Wilson

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PROCESSING - PLATING SYSTEMS - UNCLAD 2020 ALUMINUM ALLOY--

CHEMICAL AND PHYSICAL PROPERTIES - EVALUATION OF -

PURPOSE:

The use of X-2020 aluminum, a new alloy possessing a high strength to weight ratio at elevated temperatures, is being considered for applications on the B-58. Through its use, an appreciable weight saving over conventional aluminum alloys might be realized.

One of the criteria in the selection of this material is its electroplating characteristics and electroplated properties. The purpose of this test was to evaluate the plating methods and plate properties of various plating systems for unclad X-2020 aluminum alloy exposed to elevated and room temperature heat soak treatments.

SUMMARY:

Specimens of X-2020 aluminum alloy were plated by procedures employing (1) no pretreatment of basis metal and (2) sodium zincate pretreatment of basis metal. The specimens were plated with chromium and tin by standard electrolytic methods and with electroless nickel by the Alkaline type, modified Hydrac type, and Dow Process type baths. Plated specimens were then exposed to 100 hour heat soak performance tests at temperatures up to 350°F and subsequently evaluated for plating adhesion and corrosion resistance.

Two general classes of specimens were prepared: (1) coupon type for tape stripping and bend adhesion tests and for salt spray corrosion tests, and (2) galvanic specimens for use in three phase JP-4-- salt water immersion tests. The galvanic specimens consisted of plated X-2020 aluminum coupled to coated HK-31 magnesium by procedures specified in FPS-0060.

Visual observations and tape stripping test results revealed that the platings applied to X-2020 aluminum by the direct method (no pretreatment) exhibited extremely poor adhesion. Therefore, the direct plated specimens were not evaluated further.

SUMMARY: (continued)

The results of bend and tape stripping tests indicate acceptable adhesion for specimens prepared by all plating procedures employing the zincate pretreatment. No detrimental effects in adhesion were noted as the result of the elevated temperature heat soak performance tests.

The salt spray corrosion resistance was within the respective specification requirements for all platings employing zincate pretreatment with the exception of those of chromium and tin exposed to the 350°F heat soak.

All galvanic corrosion test specimens exposed to three phase immersion failed within 48 hours except those incorporating tin plated components that were held at room temperature.

PROCESSING - PLATING SYSTEMS - UNCLAD 2020 ALUMINUM ALLOY -
CHEMICAL AND PHYSICAL PROPERTIES - EVALUATION OF -

OBJECT:

- (1) To establish methods for plating tin, chromium, and Alkaline Type, Hydrac, and Dow process electroless nickel on X-2020 aluminum.
- (2) To evaluate the adhesion and corrosion resistance of the above plates on X-2020 aluminum after exposure to room and elevated temperatures.

TEST SPECIMENS, MATERIALS, AND EQUIPMENT:

A detailed list of the test specimens, materials, and equipment used during this test is given in Table I. Test specimens for plate adhesion and salt spray corrosion tests were 1" x 5" x .040" coupons of bare X-2020 aluminum alloy. Galvanic corrosion specimens were formed by coupling similar coupons (plated) to Dow "17" treated and painted HK-31 magnesium components which were 4" x 5" x 0.064" in size. Countersunk holes for AN-426B-4-5 rivets used for joining specimens were placed 1/4" from each end along the centerline of the components.

PROCEDURE:

An outline of test procedures, including plating methods and heat soak performance tests applicable to individual specimens are called out in Table II. Detailed procedures for pretreatments, plating, Dow "17", and paint coating operations are presented in Table III.

RESULTS:

The results of bend and tape stripping tests indicated acceptable adhesion for all specimens prepared by all plating procedures employing the zincate pretreatment method. No detrimental effects in adhesion were noted as the result of elevated temperature heat soak performance tests. Direct plating methods failed to produce the desired physical properties.

RESULTS: (continued)

The results of salt spray corrosion tests are presented in Table IV. Three phase corrosion tests on galvanic specimens are given in Table V.

DISCUSSION:

In view of the good results previously obtained for alkaline type electroless nickel on 7075-T6 aluminum as given in FGT-2106 (Determination of corrosion resistance of electroless nickel plated bare 7075-T6 aluminum) this process was also evaluated on X-2020 aluminum. The low Hydrac bath, also evaluated in the above test, required a similar modification (use of sulfate salts instead of the more active chloride) as reported therein. The unmodified bath reacted strongly with both 7075-T6 and X-2020 aluminum alloys, thereby preventing the formation of plates.

The direct plating pretreatment method as presented in the procedure section was not systematically evaluated because of the poor results observed during or after the plating operation such as spontaneous peeling of electrolytic (chromium, tin) plates, and removal of electroless nickel plates by tape stippling.

Results of 250 hours salt spray on plated X-2020 aluminum previously exposed to 350°F, 300°F, and room temperature are shown in Figures 1 through 5. These photographs show the general decrease in performance of chromium and tin with increasing heat soak temperature. Specimens exposed to 350°F failed salt spray within 24 hours. Comparison of 350°F results with those obtained at 300°F indicates a rather critical temperature vs. corrosion resistance transition point, thereby limiting the use of these plates on X-2020 to temperatures not above 300°F. Electroless nickel plates were not adversely affected by elevated temperatures.

Figure 6 shows the configuration of galvanic specimens and the typical results obtained after exposure to 48 hours in 3 phase (3% salt solution - JP-4 fuel - JP-4 vapor) corrosion environment.

One hundred hour exposure, as requested, was not completed due to the heavy attack noted after 48 hours. Specimen No. 8 of Figure 6 demonstrates the acceptable resistance offered by specimens incorporating tin plates which were held at room temperature. No definite variation in corrosion effects for different heat soak temperatures was noted for electroless

DISCUSSION: (Continued)

nickel, and the degree of corrosion was seen to be parallel to (but greater than) salt spray results.

Plating evaluations on X-2020 material were commensurate with the characteristics and behavior expected of more conventional aluminum alloys. Electrolytic processes on X-2020 required the zincate pretreatment, followed by deposition of a copper plate of approximately 0.0001" in thickness. This thickness of copper plate served to prevent penetration of active solutions through pores to the basis metal, causing a corrosive reaction and subsequent pitting or poor adhesion.

Electroless nickel baths, from considerations including stability (tendency toward spontaneous decomposition), ease of constituent control, plating rate, and plating appearance fall into a decreasing order of preference as follows:
(1) Alkaline type, (2) Dow Process, (3) Modified Hydrac. When considered from a standpoint of plate characteristics, i.e. adhesion to basis metal and corrosion protection, they fall into the following decreasing order: (1) Alkaline Type, (2) Modified Hydrac, (3) Dow process.

CONCLUSIONS:

- (1) Adherent and attractive plates of chromium, tin, Dow's electroless, Modified Hydrac, and Alkaline Type electroless nickel were produced on X-2020 aluminum by all procedures employing the zincate pretreatment.
- (2) Salt spray corrosion resistance passed specification requirements for all plates with exception of electrolytic methods (tin and chromium) on specimens that were exposed to the 350°F heat soak performance tests. Performance of chromium and tin were closely parallel at varied temperature exposures. Alkaline type electroless nickel was found to be superior to other procedures evaluated.
- (3) All galvanic corrosion specimens failed 3-phase immersion testing after 48 hours exposure with exception of the group incorporating tin plates held at room temperature.

TABLE I
TEST SPECIMENS, MATERIALS, AND EQUIPMENT

I. TEST SPECIMENS:

<u>Item</u>	<u>Quantity</u>	<u>Source</u>
Plating specimens .040" x 1" x 5" X-2020 Aluminum alloy	210	Dow Chemical Co. Midland, Michigan
HK-31-H 24 Magnesium-Thorium Alloy (FMS-0046) .064" x 4" x 5"	48	Dow Chemical Co. Midland, Michigan
5056 Aluminum rivet (AN 426B-4-5)	96	Convair Stock

II. MATERIALS:

Zincate immersion pretreatment solution	Prepared in Chemistry Laboratory (See Table III for details)
Copper strike plating solution	"
Rochelle copper plating solution	"
Stannate tin plating solution	"
Watt's nickel plating solution	"
Chromium plating solution	"
Dow Process electroless nickel (proprietary formulation)	"
Hydrac electroless nickel	"
Alkaline type electroless nickel	"
Dow "17" treatment solution	"
D.C. XP-214 silicone primer	NAPCO Paint Co., Houston, Texas

- TABLE I
 ((continued))

TEST SPECIMENS, MATERIALS, AND EQUIPMENT

III. EQUIPMENT:

<u>Item</u>	<u>Source</u>
0-350°F Oven	Blue "M" Electric Co., Blue Island, Ill.
Salt Spray Chamber	Industrial Filter & Pump Mfg. Company, Chicago, Ill.
Electroplating test fixture	Convair built
Vapor degreaser	Convair built
Bend tester	ONeil-Irwin Mfg. Co. Lake City, Minn.
Dermitron thickness tester	Unit Process Assemblies Inc., New York 3, N. Y.

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TABLE II
 OUTLINE OF SPECIMEN PREPARATION AND TESTING DESIGNATIONS

PLATING SYSTEM	PRE- TEST HEAT SOAK, #	TYPE OF TEST			
		JP-4 SALT SOIL CORROSION	TAPE STRIPPING	BEND TEST	SALT SPRAY
A. .0001" COPPER	1 Room TEMP	1-2	49-50	51-52	53-54
	2 100 HRS - 300°F	3-4	55-56	57-58	59-60
	3 100 HRS - 350°F	5-6	61-62	63-64	65-66
B. .0001" COPPER	1 Room TEMP	7-8	67-68	69-70	71-72
	2 100 HRS - 300°F	9-10	73-74	75-76	77-78
	3 100 HRS - 350°F	11-12	79-80	81-82	83-84
C. .0005" TIN	1 Room TEMP	13-14	85-86	87-88	89-90
	2 100 HRS - 300°F	15-16	91-92	93-94	95-96
	3 100 HRS - 350°F	17-18	97-98	99-100	101-102
D. MODIFIED HYDRAZINE ELECTROLESS NICKEL .001"	1 Room TEMP	19-20	103-104	105-106	107-108
	2 100 HRS - 300°F	21-22	109-110	111-112	113-114
	3 100 HRS - 350°F	23-24	115-116	117-118	119-120
E. AEROMARINE TYPE ELECTROLESS NICKEL .001"	1 Room TEMP	NO EVALUATION	193-194	195-196	197-198
	2 100 HRS - 300°F	IN THIS TEST	199-200	201-202	203-204
	3 100 HRS - 350°F	205-206	207-208	209-210	

Specimens # 25 thru 48, 121 thru 192 were used for direct plating evaluations

TABLE III

SPECIMEN PREPARATION AND TEST PROCEDURES

I. Pre-Plating Surface Treatments For X-2020 Aluminum:

- A. Perform standard cleaning operation with methyl ethyl ketone followed by vapor degreasing in trichloroethylene.
- B. Immerse for 1-2 minutes in 50% by volume nitric acid at 160-180°F to remove additional surface contaminants.
- C. Tap water rinse
- D. Etch for 2-3 minutes or until surface attains uniform appearance (black) in a solution of 20 oz/gallon sodium hydroxide held at room temperature.
- E. Tap water rinse
- F. Immerse at room temperature in 50% by volume nitric acid for 30-60 seconds as necessary to remove residue from operation D.
- G. Tap water rinse
- H. 1. Perform double zincate treatment for specimen so designated.
 - a. Using 2S aluminum wire suspension, immerse for 60 seconds in a solution of 70 oz/gal sodium hydroxide and 12 oz/gal zinc oxide held at room temperature. Agitate part mildly.
 - b. Tap water rinse
 - c. Remove zinc film by 30 seconds immersion in 50% by volume nitric acid at room temperature.
 - d. Tap water rinse
 - e. Repeat step "a"
 - f. Tap water rinse and perform succeeding plating operation immediately.

TABLE III
 (continued)

SPECIMEN PREPARATION AND TEST PROCEDURES

I. H. (continued)

2. Specimens designated for direct plating were processed by steps IA through IG only, then immersed for 1 minute in 5% by volume hydrochloric acid for activation, rinsed, and plated.

II. Procedure for Application of Plates On X-2020 Aluminum Alloy By Electrolytic Processes.

A. Copper strike

Composition

Copper Cyanide	2.0 oz/gal
Sodium Cyanide	3.0 oz/gal
Caustic Soda	0.25 - 0.50 oz/gal

Operating Conditions

Temperature	120°F
Anodes	Stainless Steel
Potential (volts D.C.)	6
Immerse with potential applied to part.	

B. Tap water rinse

C. Copper Plate to 0.0001" thickness

Composition:

Copper Cyanide	3.5 oz/gal
Sodium Cyanide	4.6 oz/gal
Sodium Carbonate	4.0 oz/gal
Potassium Sodium Tartrate	6.0 oz/gal
Free Sodium Cyanide	0.75 oz/gal
Sodium Hydroxide	To pH 12.6
Anodes	Pure electrolytic copper

Operating Conditions:

Temperature	130°F
Current Density	30 amps/Ft. ²

TABLE III
 (continued)

SPECIMEN PREPARATION AND TEST PROCEDURES

III. (continued)

D. Tap water rinse
 E. Application of Nickel and Chromium on System "A" Specimens, Table II.

1. Nickel Plate 0.0002" thickness

Composition

Nickel Sulfate	32 oz/gal
Nickel Chloride	6 oz/gal
Boric Acid	4 oz/gal
Anodes	pure nickel

Operating Conditions:

Temperature	140°F
Current Density	40 amps/Ft ²
pH	4.5

2. Chromium Plate 0.0005" Thickness

Composition:

Chromic Acid	53 oz/gal
Sulfate Ion (as sulfuric acid)	0.53 oz/gal
Anodes	Lead alloy

Operating Conditions:

Temperature	130-140°F
Current Density	2 amps/in ²

F. Application Of Tin (0.0005") on System "B" Specimens, Table II.

Proceed immediately from operation D to the stannate tin plating solution:

Composition:

Sodium Stannate	14 oz/gal
Sodium Hydroxide	1.25 oz/gal
Tin metal	5.6 oz/gal
Anodes	pure tin (filmed)

TABLE III
 (continued)

SPECIMEN PREPARATION AND TEST PROCEDURES

II. F. (continued)

Operating Conditions:

Cathodic Current Density (D.C.)	30 amps/Ft ²
Anodic Current Density	15 amps/Ft ²
Temperature	150 plus/minus 5°F.

III. Procedures For Application Of Plates On X-2020 Aluminum By Electroless Processes Designated As Plating Systems C, D, and E on Table II

A. System "C" - Dow Electroless Nickel - 0.001" Thickness.
 Composition and Operating Conditions: Proprietary data of Dow Chemical Corporation not to be published in Convair Reports.

B. System "D" - Hydrac Electroless Nickel - 0.001" Thickness.
 Composition and Operating Conditions:

Nickel Sulfate	4 oz/gal
Sodium Hypophosphite	1.3 oz/gal
Hydroxyacetic (glycolic) acid	3.0 oz/gal
Sodium Hydroxide	for pH adjustment
pH	4.0-4.3
Temperature	203-212°F

C. System "E" Alkaline Type Electroless Nickel - 0.001" thickness.
 Composition and Operating Conditions:

Nickel Chloride Solution (80 oz/gal)	4.0 fl oz/gal
Ammonium Hydroxide	to pH 8-10
Sodium Hypophosphite	1.0 oz/gal.
Ammonium Chloride	6.5 oz/gal
Sodium Citrate	9.5 oz/gal
Temperature	195-205°F

IV. Passivation of Plates:

System "B" - Immerse in 0.25% by weight chromic acid at 180°F for 30 seconds.

Systems "C - E" - Immerse as above for a period of 10 minutes.

TABLE III
 (continued)

SPECIMEN PREPARATION AND TEST PROCEDURES

V. Preparation of Galvanic Corrosion Test Specimens

A. HK-31 Magnesium Components:

1. Clean and degrease according to standard procedures stated in Section I of this Table.
2. Apply a Dow "17" coating as specified in FPS-0045 to a thickness of .0003".

Composition:

Ammonium acid Fluoride	32 oz/gal
Sodium Dichromate	13.3 oz/gal
Phosphoric acid 85%	11.5 fl oz/gal

Operating Conditions:

Current Density (amps/Ft ²)	5 - 50
Temperature	160-180°F
Potential (A.C.)	110 volts

3. Apply D.C. XP-214 silicone base primer to thickness of .35 plus or minus .05 mils.
4. Air dry for 30 minutes, then bake at 350°F for 30 minutes.
5. Sand lightly with No. 400 "wet or dry" sandpaper to remove protrusions.
6. Apply second coating of XP-214 primer as specified.
7. Repeat step 4.

B. Plated X-2020 Aluminum Components: Preparation procedures previously specified in this table.

C. Joining of Aluminum and Magnesium Components:

Specimens were joined with AN 426B-4-5 rivets by procedures specified in FPS-0060, Corrosion Protection and Finishing of Magnesium - Thorium and Dissimilar Metals.

TABLE III
 (continued)
SPECIMEN PREPARATION AND TEST PROCEDURES

VI. Pre-test Heat Soak: Specimens to be subjected to 100 hours heat soak at 350°F, 300°F, and those to be held at room temperature are specified in Table II.

VII. Evaluation of Specimens:

A. Tape Stripping Adhesion Test: Pressure sensitive tape 3M No. 250 was applied uniformly to the plated specimens designated in Table II. Adhesion testing, was accomplished by removal of tape with one abrupt motion, followed by examination of the tape and specimen for evidence of plate adhesion failure.

B. Bend Test For Plate Adhesion: Specimens were bent through an angle of 180° on a 1/4" diameter until fracture of the basis metal occurred. Visual examination at the stressed and fractured area was made for non-adhesion of plate to determine failure.

C. Salt Spray Porosity Test: The required specimens were subjected to salt spray exposure for 250 hours in accordance with Federal Test Method Standard No. 151, Method. 811. Inspection for excessive corrosion (as specified below) was performed at 24 hour increments.

Plate	Specification	Extent Required For Failure
Chromium on Aluminum	FPS-0028	"excessive" corrosion after 48 hours
Tin on Aluminum	FPS-0029	1 pit/in. ² after 48 hours
Electroless Nickel on Aluminum	FPS-0017	No corrosion after 48 hours.

D. 3-Phase Corrosion Test: Galvanic corrosion specimens were immersed in a corrosion inducing media of 3% sodium chloride, JP-4 fuel, and JP-4 vapors held at 140°F for 48 hours. Visible breaks in coatings and corrosive attack in any phase constituted failure.

TABLE IV
 EFFECTS OF 250 HOUR SALT SPRAY ON PLATED X-2020 ALUMINUM

TYPE OF PLATES APPLIED	PRE-TEST ENVIRONMENT #	SPECIMEN NUMBER	HOURS EXPOSED TO SALT SPRAY								CONCLUSIONS
			24	48	96	120	144	168	192	216	
.0001" COPPER .0002" NICKEL .0005" CHROMIUM	1 ROOM TEMP	53									PASS
		54									PASS
	2 100 HRS 300°F	59									FAIL
		60									FAIL
	3 100 HRS 350°F	65									FAIL
		66									FAIL
.0001" COPPER .0005" TIN	1 ROOM TEMP	71									PASS
		72									PASS
	2 100 HRS 300°F	77									FAIL
		78									FAIL
	3 100 HRS 350°F	83									FAIL
		84									FAIL
DOW ELECTROLESS NICKEL .001"	1 ROOM TEMP	89									FAIL
		90									FAIL
	2 100 HRS 300°F	95									FAIL
		96									FAIL
	3 100 HRS 350°F	101									FAIL
		102									FAIL
MODIFIED HYDRAZ ELECTROLESS NICKEL .001"	1 ROOM TEMP	107									PASS
		108									FAIL
	2 100 HRS 300°F	113									FAIL
		114									FAIL
	3 100 HRS 350°F	119									FAIL
		120									FAIL
ALKALINE TYPE ELECTROLESS NICKEL .001"	1 ROOM TEMP	197									FAIL
		198									FAIL
	2 100 HRS 300°F	203									PASS
		204									FAIL
	3 100 HRS 350°F	209									PASS
		210									PASS

TABLE V
RESULTS OF 48 HOUR 3-PHASE IMMERSION TEST ON

TYPE OF PLATE APPLIED	SPECIMEN NUMBER AND HEAT SOURCE	TYPE OF CORROSION					
		X-2020 ALUMINUM COMPONENT		HC-3 / TiAG. COMPONENT		C-10	
5 X-2020 ALUMINUM	500°C	PITS	BUSTERS	FRAYING OF PLATE	FRAYING EDGES	GENERAL APPEARANCE (CORROSION)	CORRODED APPEARANCE AT 500°C
5 X-2020 ALUMINUM	500°C	None	None	None	None	None	None
1 COPPER, 0001"	Room TEMP	Small-Numerous	None	None	Slight	RIVET AREA	HEAVY
2 TIN, 0005"	300°F	None	None	None	"	EDGE AND RIVET AREA	"
3 TIN, 0005"	300°F	ONE AREA ONLY	None	None	"	"	"
4 TIN, 0005"	100 Hrs	SIGHT	None	None	"	"	"
5 TIN, 0005"	350°F	Numerous	None	None	"	"	"
6 TIN, 0005"	100 Hrs	Small-Numerous	None	None	"	"	"
7 Room	None	None	None	Slight	None	None	PASS
8 TEMP	"	"	"	None	"	"	"
9 TIN, 0005"	300°F	"	"	"	"	RIVET AREA	FAIL
10 100 Hrs	4	"	"	"	"	END AND RIVET AREA	"
11 350°F	"	Small-Numerous	"	"	"	RIVET AREA	"
12 100 Hrs	Small-Numerous	Small-Numerous	"	Slight	Holes, Slight Edge Areas	HEAVY	"
13 Room	None	None	HEAVY	HEAVY	HEAVY	HEAVY	FAIL
14 TIN, 0005"	TEMP	"	"	HEAVY	None	"	" ON END
15 TIN, 0005"	300°F	"	"	Slight	HEAVY Edge corrosion	SIGHT	"
16 100 Hrs	"	"	"	Slight	" ON END	HEAVY ON END	"
17 350°F	1	"	"	HEAVY	" ON ONE EDGE	HEAVY	"
18 100 Hrs	2	Slight	HEAVY	HEAVY	" ON END, Holes in BASE	"	"
19 Room	None	None	None	None	None	SIGHT - / PAT	FAIL
20 TEMP	"	Medium	"	"	"	"	"
21 300°F	"	Slight	"	"	RIVET AREA	"	"
22 100 Hrs	"	Slight	"	"	5 Holes	MED	"
23 350°F	"	Slight	"	"	Holes & Heavy on Edge	Heavy	"
24 100 Hrs	"	Heavy	"	"	4 Holes, Edge Corrosion	Heavy	"

TABLE VI
PHOTOGRAPHIC INDEX

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